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CREEP AT ELEVATED TEMPERATURES AND HIGH VACUUM

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K. SCHRODER, A. GIANNUZZI AND G. GORSHA

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ABSTRACT

High temperature, low pressure creep experiments on copper and silver wires show that surface preparation can effect the creep rate markedly.

It is presently very difficult to obtain a well defined surface state of metal wires. It is therefore difficult to interpret strength changes due to modifications of environmental conditions during testing. Therefore, we tried in our experiments to test a sample in two different surface conditions. The test conditions during creep were kept constant, so that changes in creep rates are due to modifications of the surface in between deformation. The creep rate of a silver or copper wire was initially measured in a "clean" state, produced by argon bombarding, and then after oxidation. In a second series of tests, the creep rate of a blank section and a section coated with a metal film was measured simultaneously.

Tests on copper samples in the argon-bombarded state show a much higher creep rate than after oxidation in the final part of the test. This is attributed to the oxide film and "internal oxidation", because the reduction of the creep rate by a factor of up to four after oxidation is too large to be associated only with the load carrying capability of the oxide film, or its capability to act as an obstacle for dislocations inside the metal. Similar tests on silver show that its creep rate is independent of argon bombarding or annealing in air at elevated temperatures. Silver oxides are not stable at this condition. This should explain why the silver creep rate is independent of testing condition.

Measurements on iron and palladium coated silver wires show that an iron surface coating may even increase the creep rate above that of the pure

silver. Similar experiments on palladium coated silver wires lead to the expected decrease of the creep rate. These results may be related to the complete solubility of palladium in silver, whereas iron is practically insoluable in this metal.

INTRODUCTION

We studied how the surface structure of a metal effects the creep rate. Several investigations are available which show that surface preparation will effect mechanical properties. Some of these measurements were conducted in a vacuum environment. However in most of these experiments, the sample was tested in a vacuum and the results were compared with tests in an atmosphere. It is therefore possible that changes in the mechanical properties are not solely due to changes in the surface structure, but to changes in the test atmosphere. In our experiments, tests were always conducted in a vacuum, so that creep changes are due to sample modificients in between the creep measurements. These sample modifications took place in the unloaded state, in which usually air was admitted to the sample.

The first series of tests were executed on copper. A detailed description of the experimental arrangement has been given in a previous report, which contains as an appendix parts of a M.S. thesis by A. Giannuzzi describing experiments and their interpretation. The results are summarized in Appendix I. These experiments show that the creep rate of copper may be reduced through oxidation by a factor of four. This is a much larger effect than was originally anticipated. This is possible only because the surface/volume ratio is very large. The diameter of sample usually 0.01", so that the sample consisted practically only of a surface layer (A surface layer 0.0025" deep would contain 75% of the sample!).

To make sure that these results are not due to some experimental uncertainties, G. Gorsha tested silver wire of 0.010" diameter in a similar way as copper was tested. He found that the creep rate of silver is not effected by

annealing in air or by argon-bombarding. Silver oxide is only stable below 190°C. Our tests were always conducted above 190°. Therefore, our silver is without oxide films. Naturally, monolayers of oxygen on silver may still exist, but we were unable to detect the influence such layers have on the creep rate. Only in one case we found that the creep rate of silver decreased after oxidation. In this case we suspect that argon bombarding led to a sputtering of atoms of a nichrome wire on to the silver wire. This produced a metal film of unknown composition, which oxidized during annealing in air and reduced the creep rate. Appendix II contains the abstract of Mr. Gorsha's M.S. thesis which appeared as an appendix in a previous report. Appendix III is a reprint of Acta Metallurgica, which describes the experiments and interpretation of our work.

Since our last report, two new types of experiments have been conducted. The first series of creep tests was done on Sn single crystals. These measurements would eliminate effects due to grain boundary. Further, the melting point of tin is so low that one can obtain creep rates of 0.01/min at room temperature. Therefore, the test pressure can be kept much lower values than in our tests on copper and silver.

The second series of tests was conducted on copper and silver wires coated with iron or (only for silver) with palladium. We expected that such experiments would show how metallic interfaces would effect the creep rate.

Mr. R. Naster is working on this program. Presently he is teaching at a high school instead of continuing his graduate work. The completion on his M.S. thesis is therefore delayed. This thesis will be submitted in a few months as a technical report.

EXPERIMENTAL PROCEDURE

Creep tests on Sn-single crystals were conducted in essentially the same way as test on copper and silver described in annexed Appendix III.

Samples with metallic coatings were tested within a modified sample support (See Fig. 1). The partially coated wire was wrapped around the upper wire support (A), so that the blank section of the wire was parallel to the coated section. Loads of equal weight were attached to the two sections. This assured that the coated and blank wire sections were subjected to identical testing conditions and annealing procedures. Differences in creep rates should be due only to the surface films, which were prepared by evaporation in varian vacuum system at pressures of about 10^{-5} Torr. Modification in the length change monitoring system improved the accuracy of measurements to about 0.01% for measurements reproduced in Figures 6 to 11.

EXPERIMENTAL RESULTS AND DISCUSSION

Tests on tin (Fig. 2-4) gave relatively larger scatter. Within experimental accuracy, the creep rate is the same before and after argon bombarding. This is not surprising. The appearance of the sample surface did not change during argon bombarding, indicating that we were not able to remove completely the surface oxide films.

Figures 5 to 7 show results obtained on copper wires of 0.01" diameter. The decrease in scatter of data points in Figures 6 to 11 is due to a change in experimental procedure. The measurements show that the major change in creep rate between the coated and uncoated section of the wire occurs predominately in the primary part of the creep curve. The reduction in the creep rate due to coating was expected. Not only would the thin film carry a fraction of the load, but it would strengthen the oxide film on the copper, so that the previously proposed model to explain the reported reduction in creep rate due to oxidation would be confirmed.

One has to keep in mind that the copper wire section without iron film has a similar or the same oxide film as the iron coated section. These samples were not cleaned by argon bombarding before iron was deposited. This indicates that the iron film affects the dislocation movement even through the oxide film.

We expected a more pronounced reduction of the creep rate in iron coated silver wire, because silver has no oxide film. Surprisingly, we found that an iron film increases the creep rate of silver wire (Fig. 8 and 9). This cannot be attributed to experimental difficulties, because identical tests on silver wire with palladium films (Fig. 10 and 11) show again the expected decrease in creep rates with surface coating.

We suspect that this effect is due to the fact that palladium forms a complete series of solid solutions with silver whereas iron is nearly immiscible in silver. Presently we may only speculate on the cause of the increase of the creep rate due to an iron film. Fig. 12 shows schematically a hypothetical sample cross section. Because of the low miscibility of iron in silver, good contact between both materials is expected only in selected areas. More contact may develop during deformation. These interface areas will be relatively weak but they will probably not inhibit deformation. They may act as stress raisers, or they may act as a dislocation source. Both effects could lead to a decrease in strength. More experiments are required to investigate this effect and it's importance for the design of composite materials.

SUMMARY AND CONCLUSION

Creep tests on silver and copper wires at elevated temperatures in a high vacuum show that modifications of the surface structure effects the creep rate of copper and silver markedly. Oxidation, and firmly bound metallic surface films reduce the creep rate. These tests can be explained with standard dislocation models.

Silver wires with an iron surface film have a higher creep than blank wires. Further studies are required to explain this phenomena.

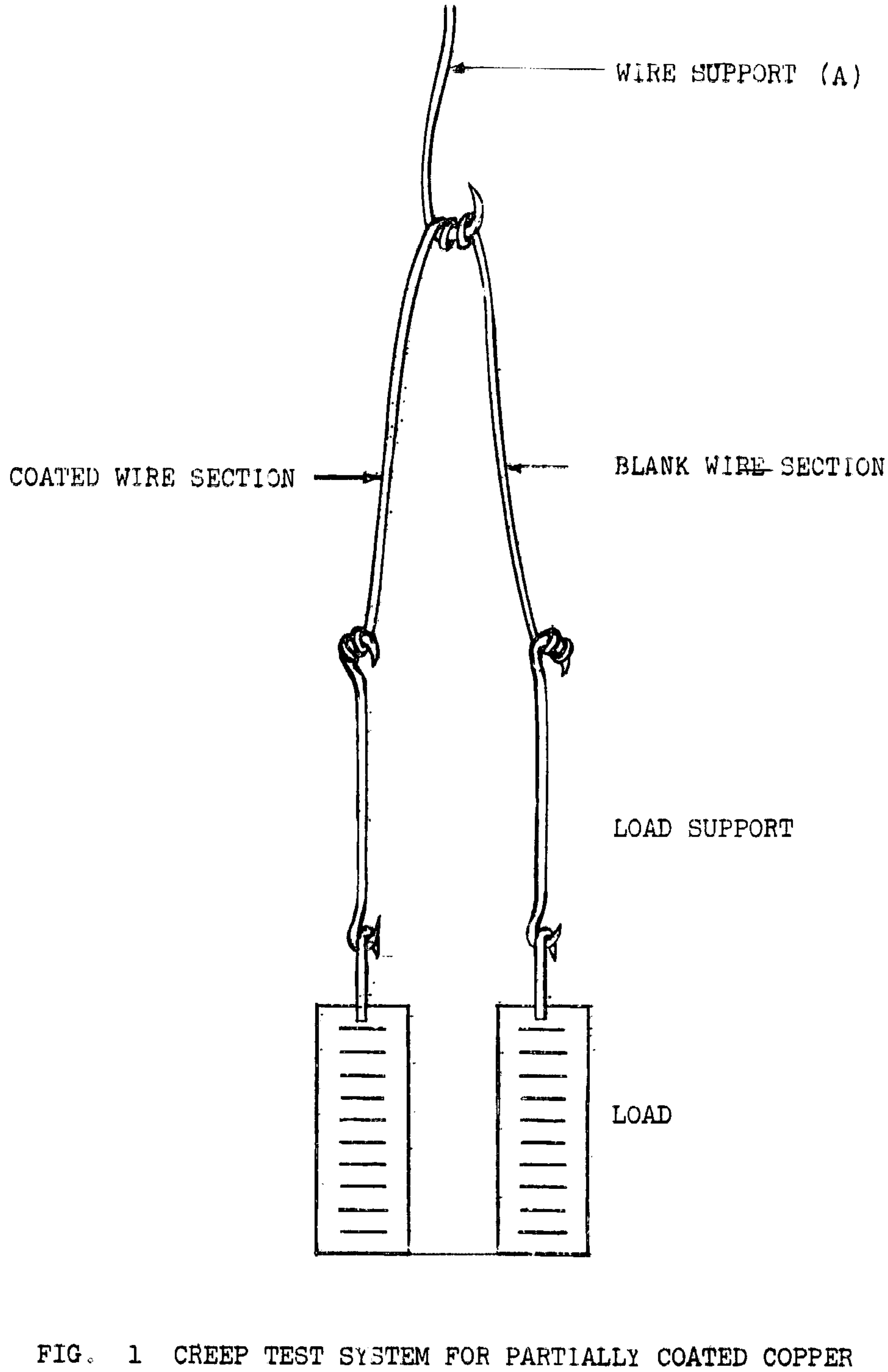

FIG. 1 CREEP TEST SYSTEM FOR PARTIALLY COATED COPPER AND SILVER WIRE

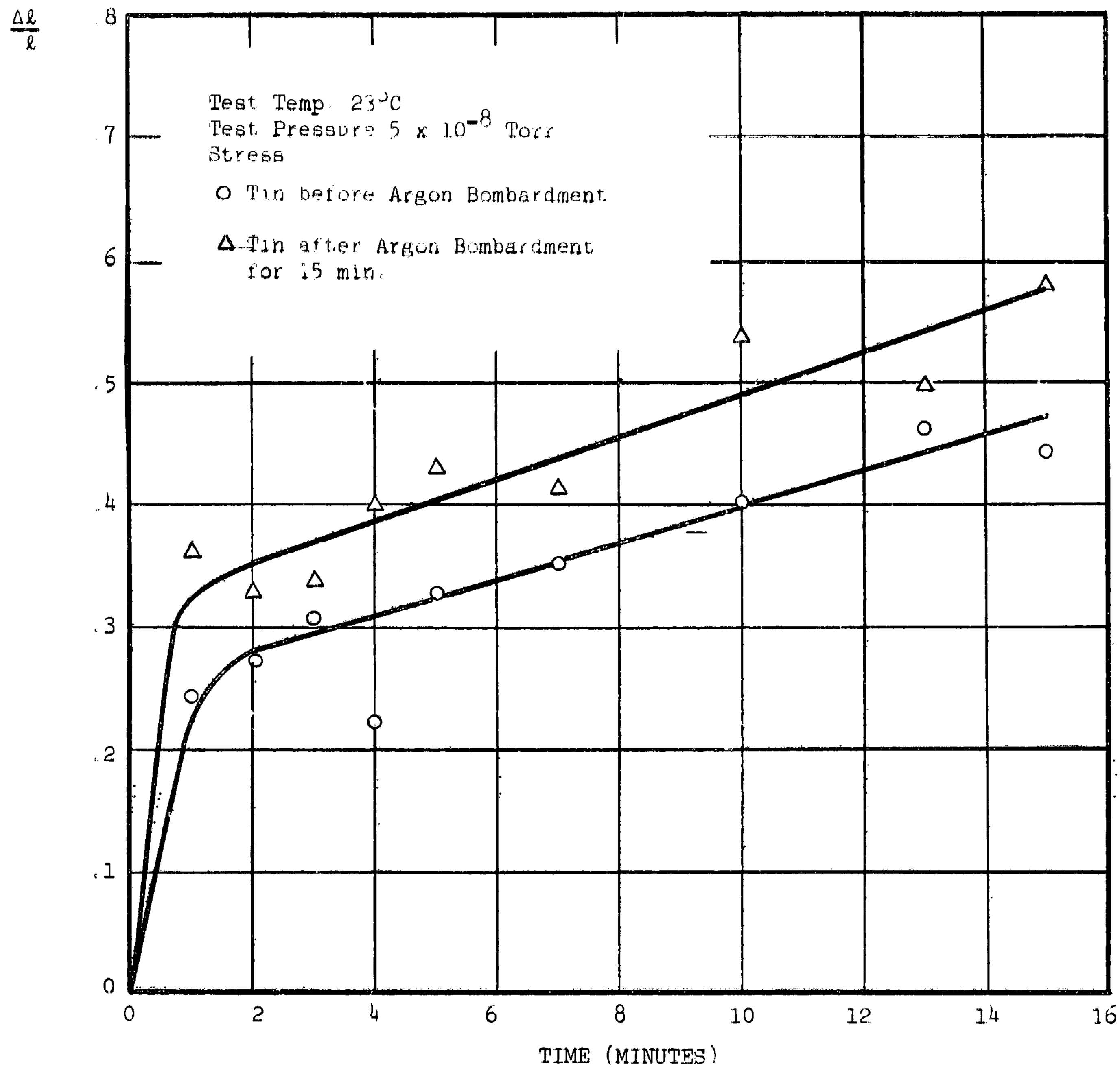

FIG. 2 CREEP TEST ON TIN SINGLE CRYSTAL

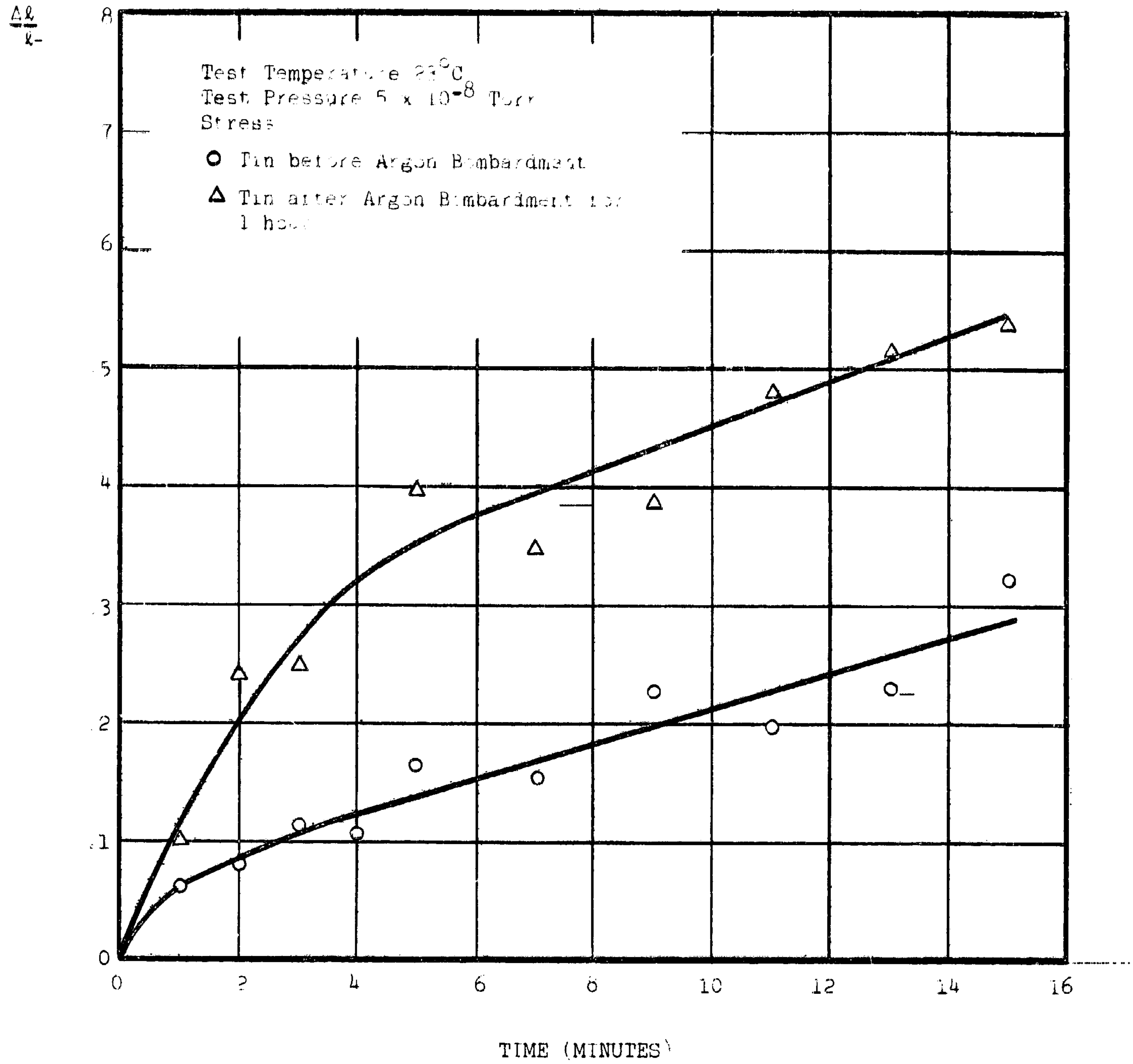

FIG. 3 CREEP TEST ON TIN SINGLE CRYSTAL

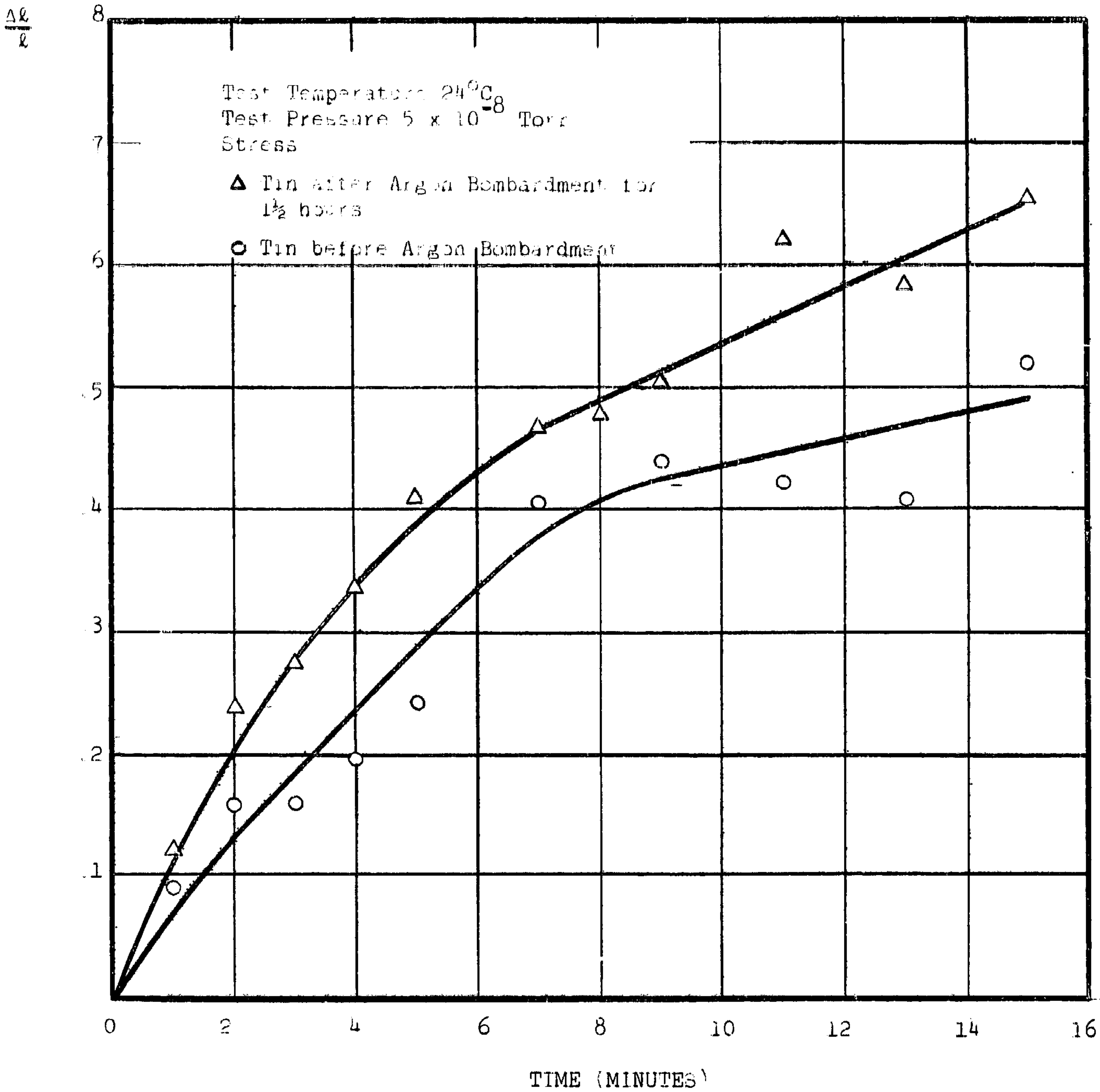

FIG. 4 CREEP TEST ON TIN SINGLE CRYSTAL

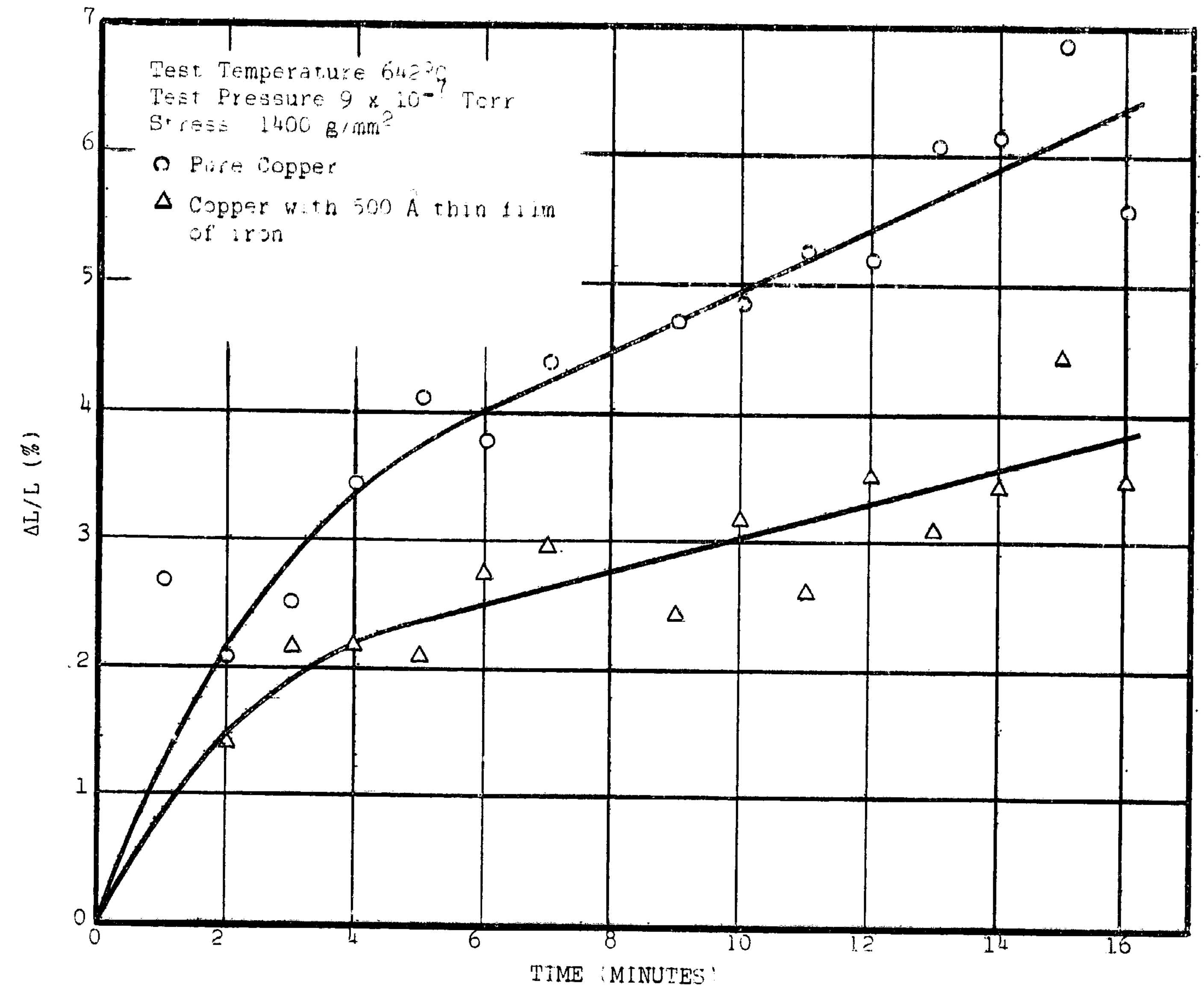

FIG 5 CREEP TEST ON COPPER WIRE WITH A DIAMETER OF 0.01"

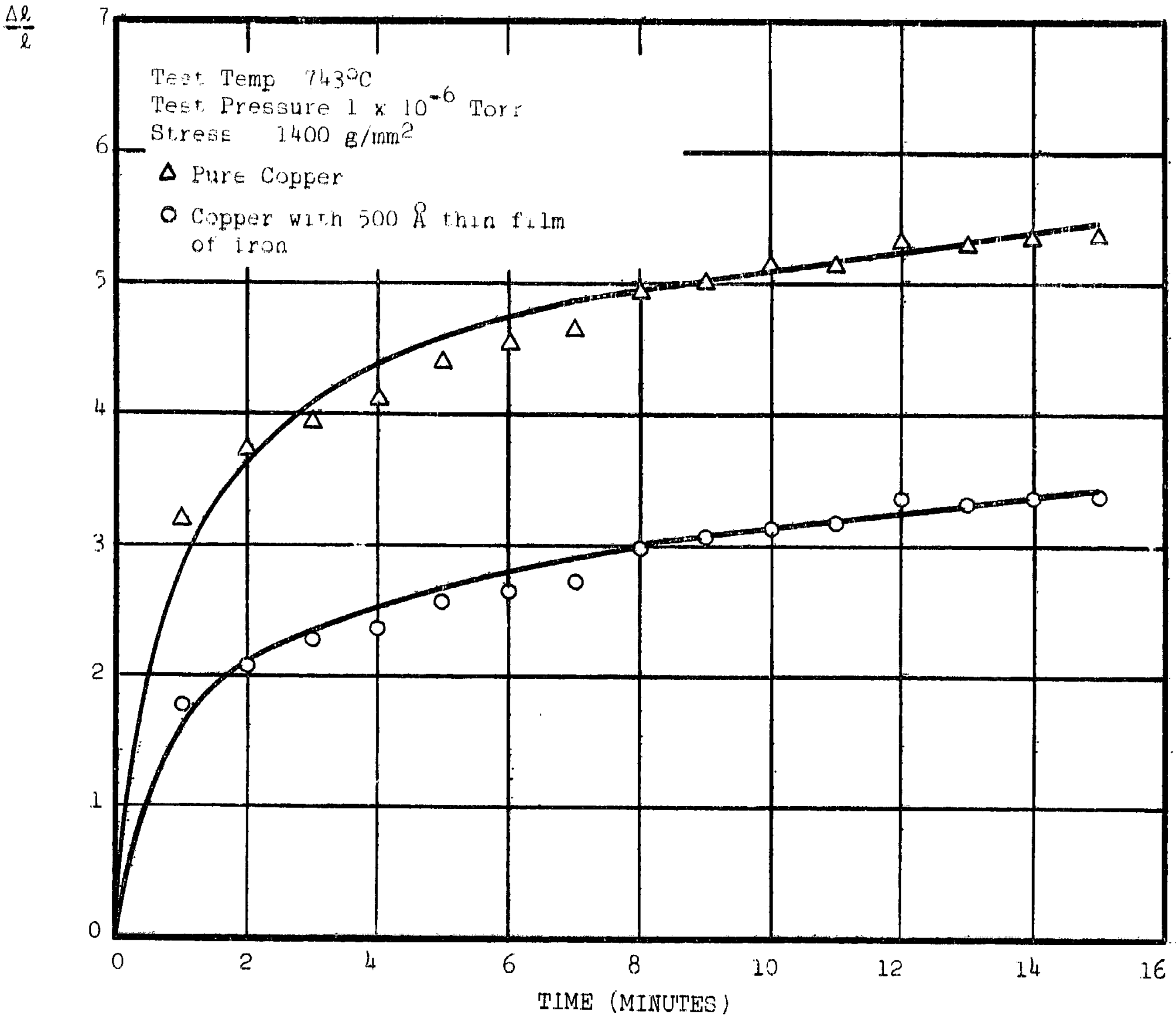

FIG 6 CREEP TEST ON COPPER WIRE WITH A DIAMETER OF 0.01"

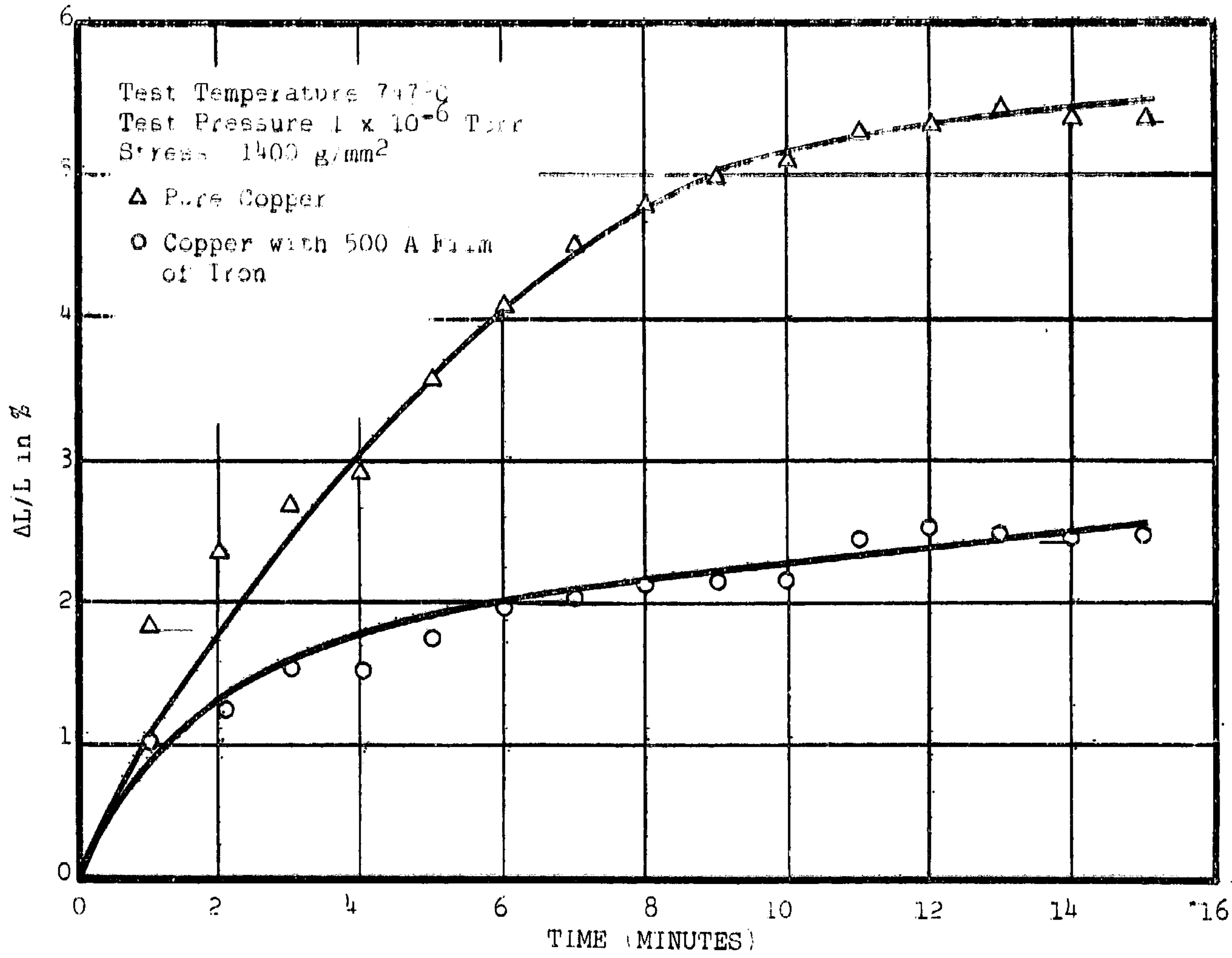

FIG. 7 CREEP TEST ON COPPER WIRE OF 0.01 DIAMETER

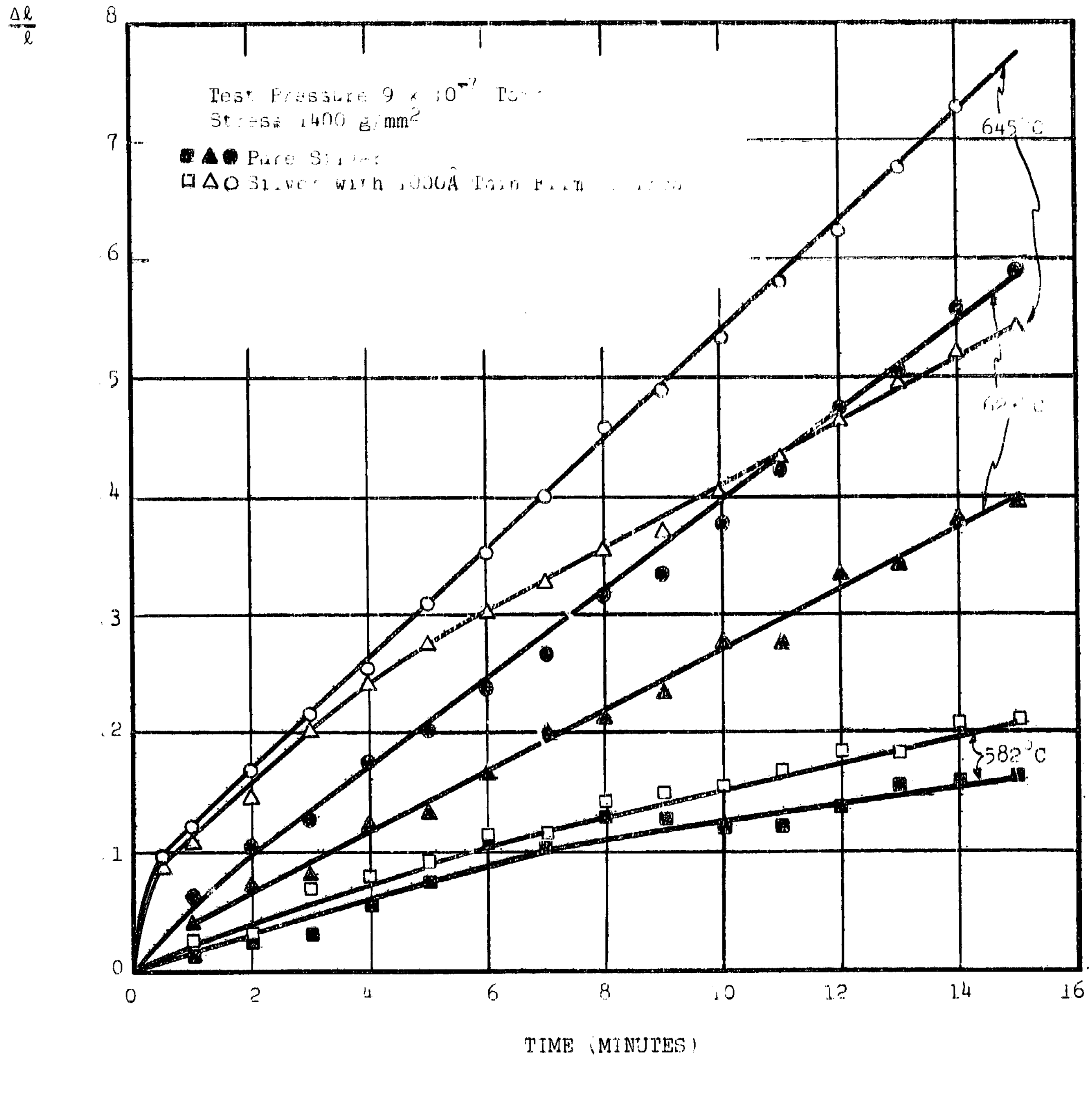

FIG. 8 CREEP TEST ON SILVER WIRE OF 0.01" DIAMETER

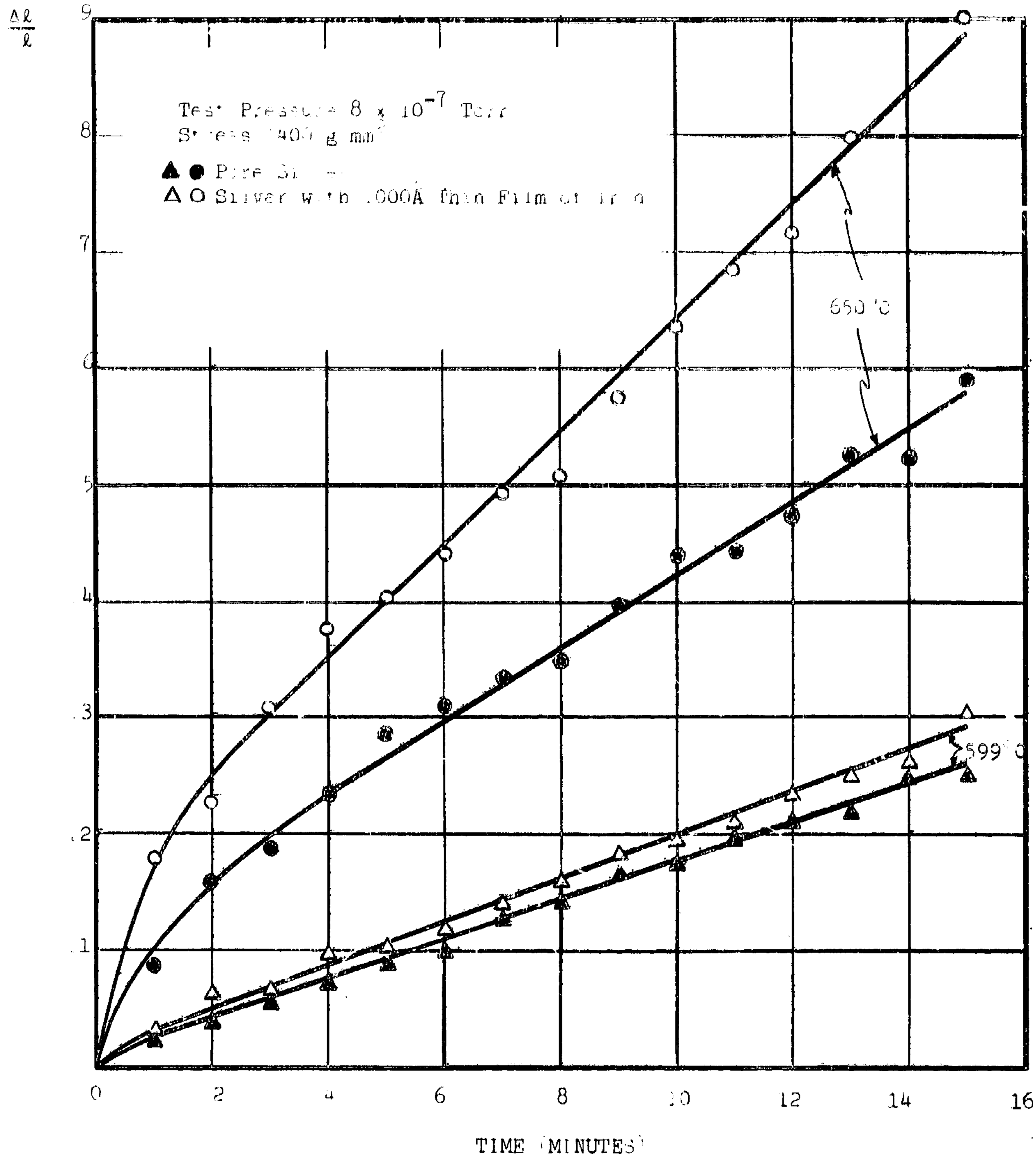

FIG 9 CREEP TEST ON SILVER WIRE OF 0.01" DIAMETER

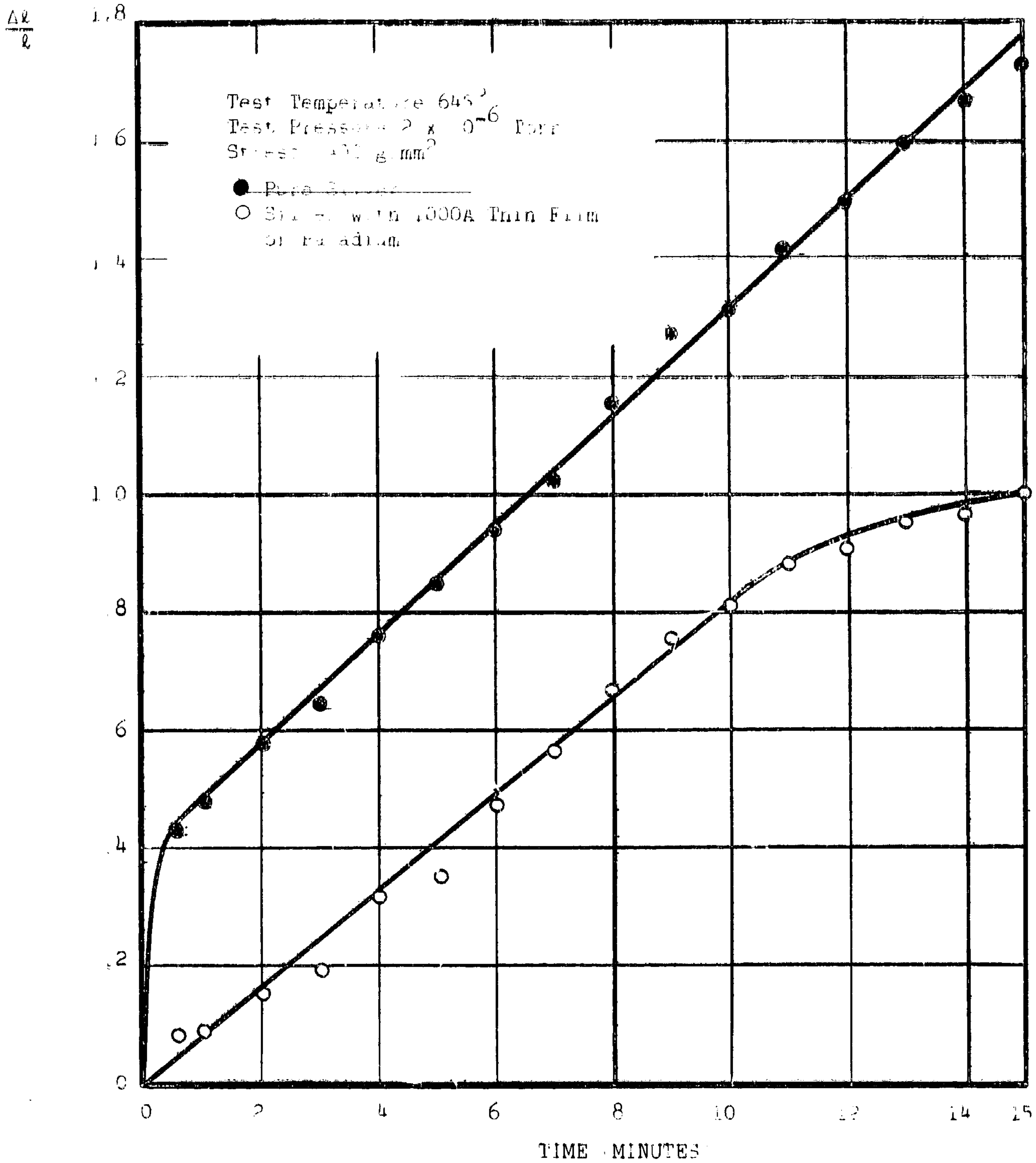

FIG. 10 CREEP TEST ON SILVER WIRE OF 0.01" DIAMETER

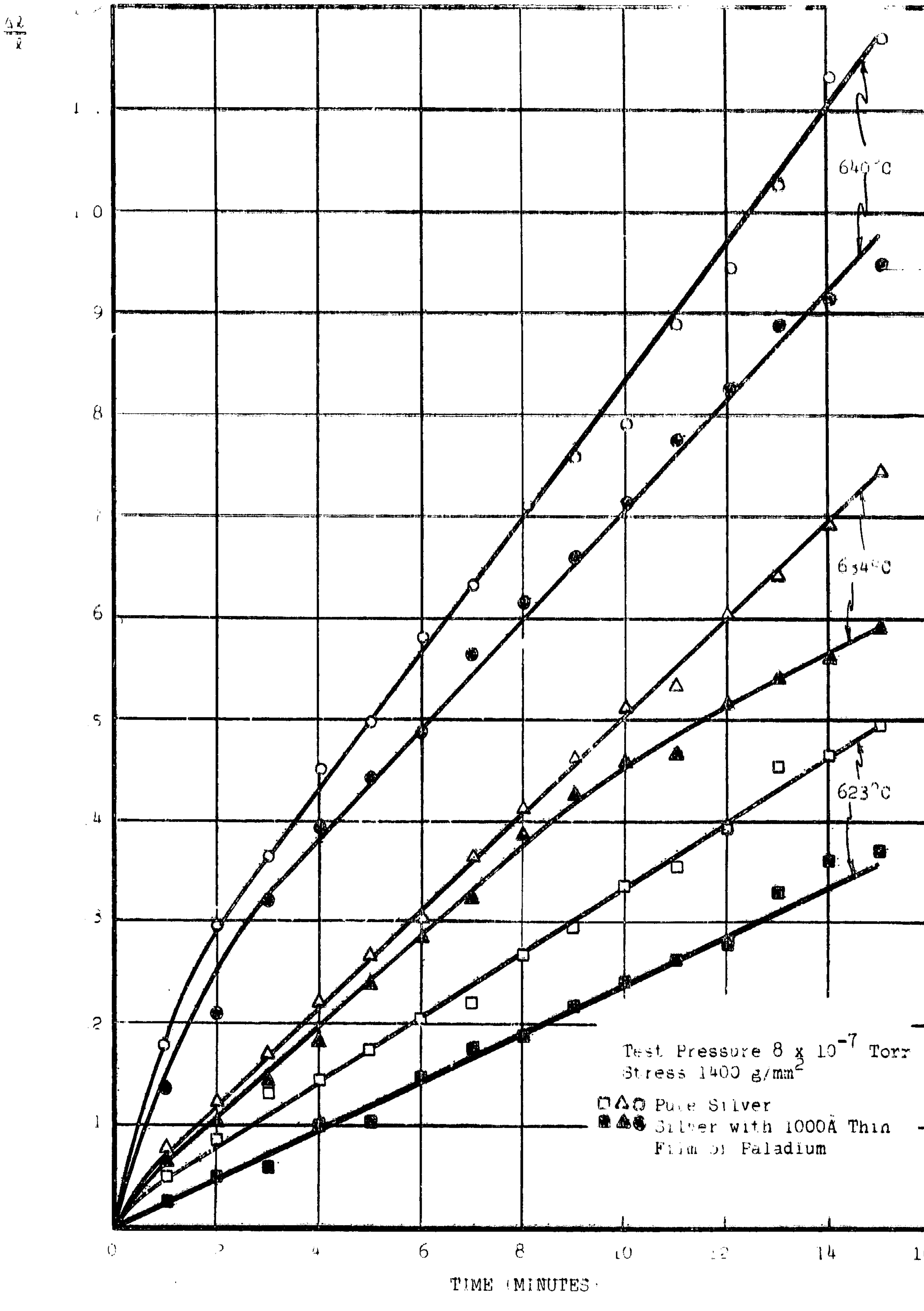

FIG. 11 CREEP TEST ON SILVER WIRE OF 0.01" DIAMETER

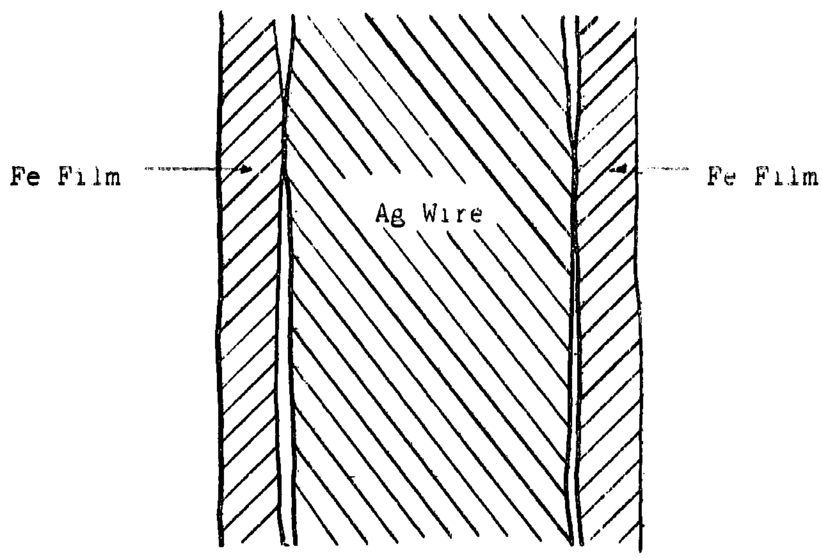

FIG. 12 HYPOTHETICAL CROSS SECTION OF SILVER WIRE WITH IRON FILM

APPENDIX I

ABSTRACT OF M.S. THESIS

by A. Giannuzzi

A series of creep tests were performed on commercial grade and high purity copper wire in high vacuum and at elevated temperatures to determine whether or not surface preparation has an effect on the creep properties of the copper.

A specimen was "cleaned" by argon ion bombardment and tested, then oxidized and tested at the same temperature and approximately the same pressure. The results indicated that the "clean" sample has a higher creep rate than does the oxidized sample.

It is presently not possible to propose a detailed dislocation model to explain the data. However, the results may be explained in part in the following manner. The creep rate of the oxidized sample is lower than for the "clean" sample because dislocations may pile up at the oxide surface or because of oxygen diffusion to grain boundaries and to dislocations. This may produce a more brittle grain boundary, an increase of frictional forces on dislocations or the oxygen may inhibit the operation of dislocation sources.

APPENDIX II

ABSTRACT OF M.S. THESIS

by G. Gorsha

A series of high temperature creep tests were performed in a high vacuum on high purity (99.999%) polycrystalline silver wire to determine whether or not an anneal in air has an effect on the steady state creep rate of the silver. The specimens were "cleaned" by argon ion bombardment and creep tested, then annealed in air and retested. No change in the minimum creep rate of silver was found.

Since silver supports no oxide at these temperatures, these results helped to confirm the theory that oxide surface films are the cause of decreases in creep rates observed in analogous tests on copper which does support an oxide surface film. The theory states that oxide films on the surfaces of metals inhibit the egress of dislocations causing them to pile up in a "debris layer" which produces backstresses on the dislocation sources thereby lowering the creep rate of the metal.

The results are particularly important to space vehicle design since space environment clears the surfaces of exposed materials.